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Fabrication of the Textured Ni-9.3at.%W Alloy Substrate for Coated Conductors

M. M. Gao, H. L. Suo, J.-C. Grivel, Y. Zhao, P. K. Gao, M. Liu, and L. Ma

Abstract—It is difficult to obtain a sharp cube texture in the Ni-9.3at.% W substrate used for coated conductors due to its low stacking fault energy. In this paper, the traditional cold rolling procedure was optimized by introducing an intermediate recovery annealing. The deformation texture has been improved after three recovery annealing cycles at 500°C for 2 h during the cold rolling process. After optimized recrystallization annealing, a Ni-9.3at.% substrate with a cube texture content of 87.7% ($< 10^\circ$) has been obtained. The effect of the intermediate annealing on cube texture formation is attributed to the improved deformation texture and to the enhanced pre-existing cube oriented structure in the deformed matrix.

Index Terms—Coated conductor, cube texture, intermediate annealing, substrate.

I. INTRODUCTION

FOR the Ni-based alloys employed for fabrication of the metallic textured substrates, an excellent recrystallization cube texture and a non-ferromagnetic behavior at the application temperature of 77 K are the basic requirements for the epitaxial growth of buffer/YBCO coated conductors, and are expected to eliminate the magnetization losses in alternating current applications. Furthermore, a sufficiently high tensile strength at elevated preparation temperature is also necessary to ensure continuous multi-step reel to reel deposition processing. Thus, the Ni-9.3at.%W (Ni9W) alloy is of great interest due to its high yield strength and non-ferromagnetic properties at the operating temperature (77 K) of coated conductors. Nevertheless, a perfect sharp cube texture is very difficult to produce because of the high W content, resulting in a drastic decrease of the stacking fault energy (SFE). The deformation texture has been changed from copper-type (pure Ni) to Brass-type (Ni9W) [1], [2]. The

relevant characteristics of a metallic substrate which can produce the sharp texture is the existence of the Copper-type deformation texture which is dominated by C and S orientations. Note that the main effect of the cube texture formation in Ni9W alloy is to inhibit the deformation texture transition, and thus to increase the intensity of C and S orientation which are related to the nucleation frequency and the grain growth of cube texture.

The origin of cube texture formation in face-centered cubic (fcc) materials has been developed and debated for several decades, and the opinion has focused on two alternative models, oriented nucleation (ON) and oriented growth (OG) [3]–[5]. According to the ON mechanism, the origin of cube texture lies in the preferred nucleation of cube grains from pre-existing cube-oriented structure in the deformed materials. In contrast, the cube texture development is attributed to the selective growth of grains with specific orientation. In order to obtain a sharp cube texture in Ni9W alloys, several processing techniques have been employed to increase the stacking fault energy or to optimize the deformation texture and pre-existing cube oriented structure in the deformed matrix. Zhou Y.X. *et al.* [6] have improved the cube texture content to 90% ($< 10^\circ$) by powder metallurgy, due to the high energy ball milling which can increase the SFE of the Ni9W alloy. Eickemeyer J. *et al.* [7] have found out that the chemical homogeneity of the alloy and stress relief annealing during cold rolling process is beneficial for strengthening the cube texture in the Ni-W alloy ($> 7\text{at.}\%W$) with medium to low SFE. Furthermore, the deformation texture of Ni9W alloy can be optimized to a more copper-type deformation texture by warm rolling, therefore increasing the cube texture to 93% [8]. In our previous work [9], a special rolling technique (referred to as the intermediate recovery annealing during the cold rolling process) has been successfully carried out to form a sharp cube texture in Ni-7at.%W alloy substrate.

In the present work, the intermediate recovery annealing has also been employed to strengthen the cube texture in Ni9W substrates. Due to the lower SFE compared to Ni7W alloy, more annealing cycles during cold rolling (multi-cycle recovery treatment) have been performed in Ni9W alloy tapes to produce the sharp cube texture. The evolution of deformation texture and cube texture has been investigated through the recovery cycles.

II. EXPERIMENT

The electrolytic nickel pieces (99.95%) and W bulk pieces (99.99%) were melted in an induction furnace and then cast to a cylindrical ingot. After hot forging and hot rolling, the Ni9W alloy slab has been processed by heavy cold rolling. In order to strengthen the cube texture, the intermediate recovery treatment has been carried out during the rolling process. Thus, both the single recovery treatment and the multi-recovery treatment have been inserted into the cold rolling processes. For one time

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TABLE I
THERMO-MECHANICAL ROLLING PROCESSES OF THE MULTI-RECOVERY
ROLLING TECHNIQUE IN Ni9W TAPES

Recovery Times	Thermo-mechanical rolling processing (99% total rolling reduction)
One	60% cold rolling (500 °C/2h)+97.5% cold rolling;
Two	60% cold rolling (500 °C/2h)+60% cold rolling (500 °C/2h) +93.75% cold rolling;
Three	60% cold rolling (500 °C/2h)+60% cold rolling (500 °C/2h)+ 60% cold rolling (500 °C/2h)+84.4% cold rolling;
Four	60% cold rolling (500 °C/2h)+60% cold rolling (500 °C/2h)+60% cold rolling (500 °C/2h)+60% cold rolling (500 °C/2h)+60% cold rolling ;

recovery treatment, the intermediate annealing performed using the same processing parameters with the Ni7W alloy, i.e., after 90% cold rolling reduction, the sheet was annealed at 500°C for 2 h followed by additional 90% cold rolling to a final thickness of 90 μm [9]. Table I shows the detailed thermo-mechanical parameters of the multi-recovery annealing experiment. The recovery was carried out one, two, three and four times after every 60% cold rolling reduction at 500°C for 2 h for each specimen, respectively. The starting thickness is calculated based on the thickness at the last annealing. For each rolling process, the total rolling reduction was 99%, the pass reduction being below 5%.

The deformation texture was investigated with a X-ray texture goniometer as well as with a SEM, equipped with an electron back scattering diffraction (EBSD) detector. The rolled Ni9W tapes were heat treated by a two-step annealing process [10] in a flowing Ar – 4%H₂ atmosphere (i.e., annealed at 1100°C for 60 min following treatment at 700°C for 30 min) in order to get the desired orientation. The recrystallization texture was characterized by EBSD; the measurement area was 800 μm \times 800 μm , while the scanning step size was 2 μm .

III. RESULTS

A. Effect of the Single Recovery Annealing Cycle on the Cube Texture Formation in Ni9W Tapes

Due to the efficiency of the single recovery treatment on sharpening the cube texture in Ni7W substrate [9], the formation of the recrystallization cube texture in Ni9W tapes was firstly carried out using the same rolling parameters as in Ni7W substrates, i.e., annealing at 500°C for 2 h after 90% cold rolling followed by an additional 90% cold rolling reduction.

Fig. 1 shows the (111) pole figures of the deformed Ni9W substrate without (Fig. 1(a)) and with (Fig. 1(b)) the intermediate annealing. It can be seen that these two substrates have the same deformation texture, i.e. both of them are dominated by the Brass orientation, which is the typical preferred deformation orientation for the metal or alloys with low SFE. Thus, no variation of deformation texture derived from intermediate recovery treatment is observed. However, the maximum orientation strength in the substrate with intermediate annealing has been decreased slightly. This could be caused by the additional cold rolling reduction (90%) in the substrate with intermediate annealing compared with 99% rolling reduction in the substrate without intermediate annealing.

To investigate the influence of the single intermediate annealing cycle on the cube texture formation, the as-rolled Ni9W

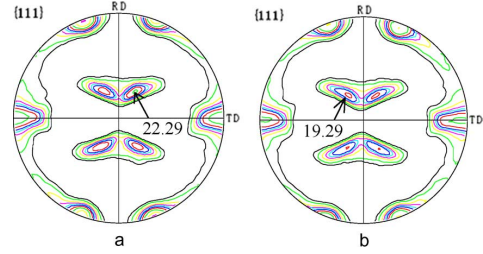


Fig. 1. The (111) pole figures of the deformed Ni9W substrate without (a); with intermediate recovery treatment (b).

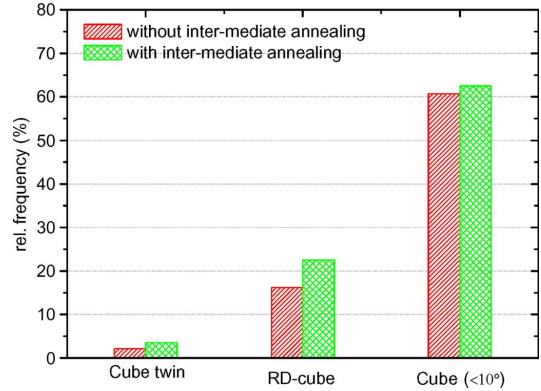


Fig. 2. Recrystallization texture components of the Ni9W substrate with and without intermediate annealing.

substrates with and without recovery annealing were annealed at 1100°C for 60 min using the two-step annealing method. Fig. 2 shows the main recrystallization texture contents in the recrystallized Ni9W substrates. Here, the tolerance angles of Cube {001}<100>, Cube twins {122}<212> and RD rotated cube {025}<100> are defined as 10°, 10° and 8° respectively. It is found that the cube texture content is 60.7% and 62.5% for Ni9W substrates, both without and with intermediate annealing, respectively. This means that the effect of single annealing cycle on the cube texture formation in Ni9W substrates may not be efficient. Meanwhile, similar results were observed for cube twinning and RD-rotated cube texture. Furthermore, the small content differences between the three recrystallization textures are probably caused by measurement errors. Thus, it can be concluded that the used rolling parameters do not lead to the expected improvement of the cube texture formation in Ni9W substrate.

B. Effect of Multi-Recovery Annealing on the Cube Texture Formation in Ni9W Tapes

To produce more cube texture in Ni9W substrates, the rolling process should be optimized to favor the cube texture formation. Due to the lower SFE in Ni9W compared to Ni7W alloy, the multi-recovery annealing technique has been considered as favorable, while the cold rolling reduction prior to the recovery treatment has been decreased from 90% to 60%.

Fig. 3 shows the deformation texture of the Ni9W substrates with different intermediate recovery cycles. It is found that both the C {112}<111> and S {123}<634> orientation increased with the number of annealing cycles, while the S orientation decreased to a certain extent in the substrate with four intermediate annealing cycles when compared with that in the three

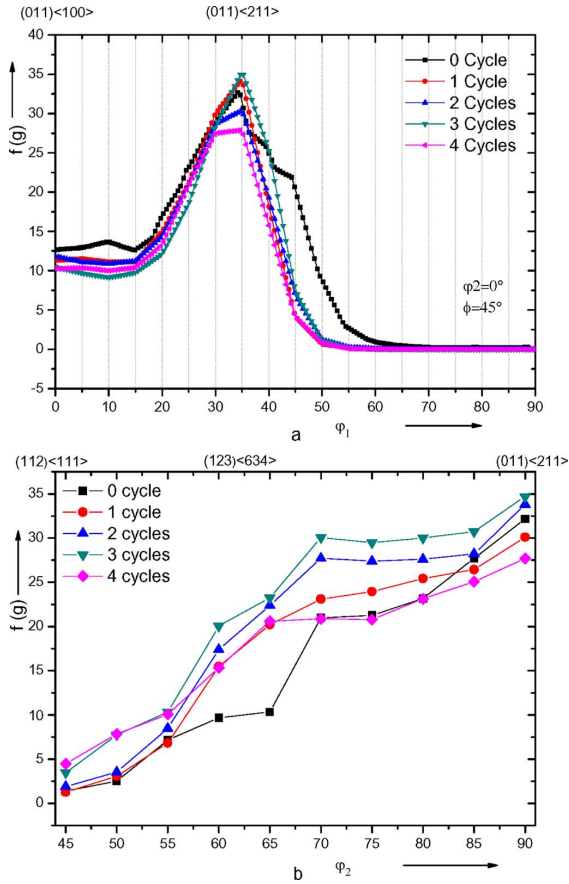


Fig. 3. The α -fiber (a); and β -fiber (b) of the deformed Ni9W substrates with different recovery annealing cycles during cold rolling.

cycle treatment. At the same time, the Goss $\{011\}\langle 100 \rangle$ orientation decreased. This is encouraging because both the C and S orientation are beneficial for the formation of cube texture on recrystallization. However, the Br $\{011\}\langle 112 \rangle$ orientation does not seem to depend on the number of recovery cycles. Nevertheless, it can be concluded that the deformation texture has been improved by the intermediate recovery during the cold rolling process.

The cube texture content of the Ni9W substrate annealed at 1100°C for 1 h using the two-step annealing method is presented in Fig. 4 as a function of the number of recovery cycles. It is clear that the recrystallized cube texture increased with increasing the number of intermediate treatments up to three recovery cycles, but decreases slightly or keeps the same level in the substrate with four cycles of intermediate recovery annealing. This indicates that the cube texture formation is not monotonically related to the number of recovery cycles. The highest cube texture content (within a misorientation angle of 10°) is 76%, which is 16% higher than that of a traditional cold rolled substrate. Therefore, it is deduced that the multi-recovery treatment favors the formation of the cube texture in Ni9W alloy.

To further strengthen the cube texture in the Ni9W substrate, the three-step annealing method [11] in which a recovery annealing step is added before the first annealing step in the two-step annealing method has been employed to anneal the substrate. After a three step annealing, the cube texture content of

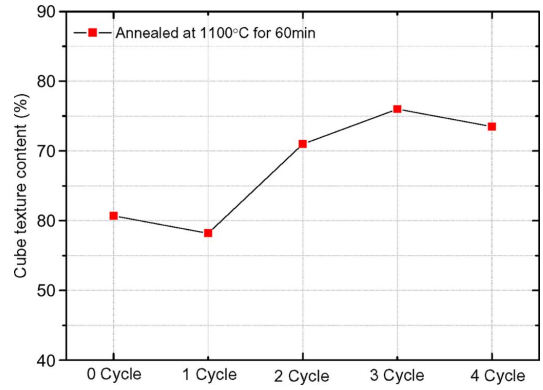


Fig. 4. The cube texture content as a function of the number of recovery cycles in the annealed Ni9W substrates.

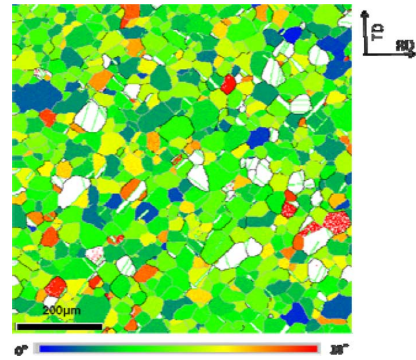


Fig. 5. EBSD map of the Ni9W substrate annealed at 1100°C using the three-step annealing method.

the Ni9W substrate can reach 87.7% ($< 10^\circ$) (Fig. 5), which is an exciting result for an alloy with such a low SFE. It is reasonable to deduce that the multi-cycle recovery annealing is a promising technique to produce sharp cube texture for satisfying the requirement of epitaxial growth in the Ni9W alloy substrate.

IV. DISCUSSION

The formation of cube texture in fcc metals can be explained by the ON and OG theories [3]–[5]. According to these theories, the cube texture is preferentially formed in the Copper-type deformation texture. In the present work, the deformation texture has been influenced by the number of intermediate annealings. The ratio (R) has been calculated between the sum of C and S texture contents and the content of 2Br texture [2], the value characterizing the deformation texture transition from copper-type to Brass-type deformation texture. To estimate the deformation texture dependence on the number of recovery cycles, Fig. 6 shows the R value as a function of the number of recovery cycles. It is found that the R value increases with the number of recovery cycles, which indicates there is more copper-type deformation texture in the recovered substrate. This is also observed in Aluminum alloys after additional annealing [12]. For the Ni9W alloy, the additional copper-type texture could be attributed to the released work-hardening during intermediate recovery, which is related to the deformation texture transition in low SFE metal or alloys [13]. However, the R value did not continuously increase after four recovery cycles. This is probably caused by the low additional cold rolling reduction

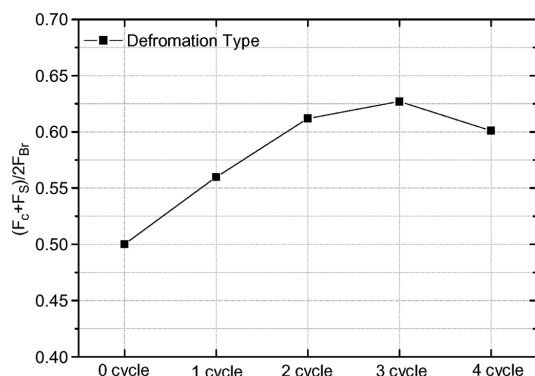


Fig. 6. Value of R as a function of the number of recovery cycles.

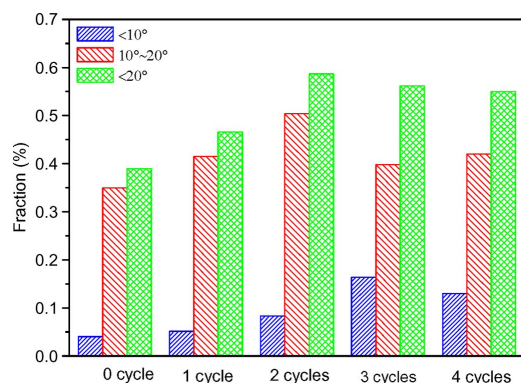


Fig. 7. The cube-oriented texture in deformed Ni9W substrates, depending on the number of recovery cycles.

(60%) after the last annealing treatment which is not sufficient to develop the deformation texture well. Indeed, this result could explain the decrease of the recrystallized cube texture in the substrate with four recovery cycles (Fig. 4).

On the other hand, the cube texture formation is also related to the pre-existing cube-oriented structure in the deformed matrix according to the ON theory. The pre-existing cube oriented structure furnishes the original nucleation sites to form the cube texture because it can undergo rapid static and dynamic recovery due to its unique dislocation arrangement [14]. In Fig. 7 the cube texture ($< 0^\circ$) and near cube texture ($< 20^\circ$) have been represented in the deformed Ni9W substrate for various numbers of recovery annealing cycles. It can be seen that the cube texture and near cube texture are increasing with increasing recovery cycle number, up to $n = 3$. This is probably due to that the recovered cube structure after one cycle annealing has to suffer heavily additional cold rolling (97.5% of the substrate with one cycle treatment) process that perhaps splits or rotates some of the recovered cube grains [15]. Thus, the inclusion of more recovery cycles in the cold rolling process is necessary for the recovery the splitted or rotated cube structures. Therefore, a higher amount of cube structure can survive up to the final deformed structure with the increasing number of recovery cycles until the additional cold rolling could not split or rotate the recovered cube structure. But there is a limit of the final additional cold rolling reduction to ensure the size advantage of the cube oriented structure compared with other orientation structures, due to its higher stability during the rolling process [16]. From Figs. 4 and 7, it can be concluded that the optimum treatment

consists in carrying out three recovery cycles during the cold rolling process.

V. CONCLUSIONS

In this work, a multi-recovery annealing process has been carried out during traditional cold rolling process, the recrystallized cube texture being strengthened upon recrystallization. The following conclusions are drawn:

- 1) The single recovery cycle which has obviously improved the cube texture formation in the Ni7W substrate is not suitable for Ni9W substrate due to the lower SFE;
- 2) A sharp cube texture (87.7%) has been obtained using a rolling technique with three recovery cycles and optimized three-step annealing method;
- 3) The mechanism of strengthening the cube texture formation by intermediate anneals relies on the improvement of the copper-type deformation texture as well as the increase of the pre-existing cube oriented structure in deformed matrix;
- 4) Multi-recovery technique is a promising solution to produce sharp cube texture in Ni-W alloy with high W contents.

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